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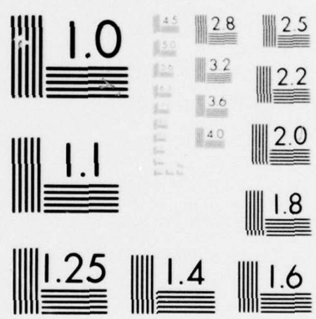

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EDGEWOOD ARSENAL CONTRACTOR REPORT

ED-CR-76106

ENZYME ALARM INSTRUMENTATION STUDIES

FIRST QUARTERLY REPORT

By

W. McAdam

R. M. Taylor

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JUL 6 1977
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October 1976

LEEDS & NORTHRUP COMPANY
North Wales, PA 19454

Contract DAAA 15-76-C-0113 new

U.S. ARMY ARMAMENT COMMAND

DEPARTMENT OF THE ARMY
Headquarters, Edgewood Arsenal
Aberdeen Proving Ground, Maryland 21010

EDGEWOOD ARSENAL

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Thermostated alarm
Foamed insulation
Controlled potential
Interference compensation
Alarm fabrication

20. ABSTRACT (Continue on reverse side if necessary and identify by block number)

Polarographic-type electrochemical enzyme alarms were designed and engineered to automatically detect C-agents in the field. The target specifications include lower power consumption, small size, light weight, and operating capability from -40°F to +120°F. Interference compensation and controlled potential circuitry developed under Edgewood Arsenal Contract DAAA15-75-C-0134 were included. Fabrication and testing of three electro-

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20. (Abstract cont'd)

chemical cells were successfully completed. Fabrication of component parts for three alarm systems is nearly complete with assembly underway.

PREFACE

The experimental and development work described in this report was authorized under Contract No. DAAA 15-76-C-0113 entitled "Enzyme Alarm Instrumentation Studies". It was performed by the Leeds & Northrup Company for the U. S. Army Edgewood Arsenal.

This report documents the part of the work performed during the period May through August, 1976. The work is continuing toward the objective of three operating prototype instruments embodying the results of the development.

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ENZYME ALARM INSTRUMENTATION STUDIES

1. BACKGROUND AND INTRODUCTION

For the past several years Edgewood Arsenal has been conducting studies on an electrochemical enzyme alarm system for the detection of chemical agents. This alarm monitors the extent of the enzyme-catalyzed hydrolysis of butyrylthiocholine iodide. In April 1975, Leeds & Northrup Company was awarded Contract DAAA 15-75-C-0134 to study means for suppressing alarm interferences with improved-time-response characteristics.

The present contract, for which this report is made, was awarded to Leeds & Northrup Company on May 13, 1976 to provide alarm instruments for limited field testing based on the improvements made under the aforementioned contract. The specific goals of this project are:

(a) Optimize hardware and circuit design of the enzyme alarm, within the time constraints imposed by goal (c).

(b) Fabricate and assemble three enzyme alarm instruments for limited field testing by Edgewood Arsenal.

(c) Deliver the three instruments to Edgewood Arsenal by November 1, 1976.

1.1 Project Plan

This project logically divides into three phases – Optimization and Design, Fabrication and Assembly, and Test and Evaluation.

1.1.1 Optimization and Design

This phase basically comprised hardware design using a combination of some approaches which had been suggested and verified by Edgewood Arsenal and the improvements resulting from Contract DAAA 15-75-C-0134. Optimization for the purposes of this contract consisted of defining the specific overall and component designs which were most suitable for fabrication, assembly, and testing within the contractual time constraints, and which could be produced with reasonable ease for additional instruments. Minimum-cost parts fabrication with manufacturing tooling was, however, not an important criterion for the designs. The major part of the design either made direct use of approaches which had been verified by Edgewood Arsenal in hand-made models,

or adapted such concepts with minimal modifications for ease in fabrication. Since testing time is severely limited under the contractual time constraints, further optimization of the Edgewood Arsenal concepts was not to be attempted.

Those parts of the alarms based on the Leeds & Northrup improvements were designed using the data available in the final report on Contract CAAA 15-75-C-0134. These latter designs were limited to the electrochemical cell and parts of the associated electronic circuitry.

1.1.2 Fabrication and Assembly

This phase consisted entirely of Model Shop fabrication of alarm parts, expediting and procurement of purchased components, and assembly of parts and components into finished alarms by engineers and technicians. This phase constitutes by far the greatest expenditure of manpower on the project, and is the most critical from the standpoint of meeting the schedule requirements.

1.1.3 Test and Evaluation

This phase is divided into two parts – tests of the first model electrochemical cell of the new design, and overall tests of the assembled enzyme alarms. The first tests of the new design cell were necessary to confirm its satisfactory operation as a basis for proceeding with construction of the alarms. These tests were accomplished during August, and are described in section 2 of this report. Overall tests of the assembled alarms will be conducted during the latter part of September prior to shipment. These will include both performance and environmental testing, and the results will be included in the Project Final Report.

Evaluation will include consideration of the alarm design as well as its performance. Final recommendations will be based in part on the evaluation.

1.2 Problems

All of the electrochemical problems associated with the design of these enzyme alarms were dealt with successfully under the previous study contract DAAA 15-75-C-0134. The problems encountered in the present work were related to adaptation of the Edgewood Arsenal concepts to designs for practical fabrication, detailed design of new parts not included in the Edgewood Arsnal concepts, and procurement of commercial components on the schedule required to meet the delivery commitment.

The successful completion of the Optimization and Design Phase, and the near completion of the Fabrication and Assembly Phase attests to the success of adapting the Edgewood Arsenal concepts and the design of the new parts. The only design uncertainty remaining is the adequacy of the new thermal insulation system used for the case. Edgewood Arsenal personnel approved the proposed new insulation system, which consists of an outer and an inner aluminum shell with the interspace completely filled with poured foaming-plastic insulation. Edgewood Arsenal's previous experience with some similar insulating systems gave reason to believe that this execution would prove adequate. However, tests of its adequcy must await the first completely assembled instrument, which will be subjected to the desired temperature range of -40° to 120°F. In the unlikely event that the insulation proves inadequate, it may be necessary temporarily to restrict the low ambient-temperature limit range for operation of the three prototypes until the insulation can be improved or the case heating increased after the first limited field test. It is understood that the first field testing will not include extreme ambient conditions, so the ambient range limitation, if required, would not preclude obtaining the desired-field-test results.

Procurement of commercial components on the required time schedule has proved to be quite difficult. Edgewood Arsenal has alleviated the problem to a considerable extent by furnishing some components which could not be purchased quickly. However, substantial expediting effort on the part of Leeds & Northrup Company has been required to obtain those components which were not available from Edgewood Arsenal. This effort has been successful, and instrument assembly is proceeding on schedule with no delays expected from commercial component shortages.

1.3 Project Status

Phase One, Optimization and Design, was completed during August. Phase Two, Fabrication and Assembly, was started about midway through the Optimization and Design Phase. Fabrication is complete, and assembly, now in progress, will be completed in September. The Test Evaluation Phase will occupy the remainder of September and October, up to the shipment date.

It is not planned that the Final Report be completed by the shipment date. Rather, it will be prepared following the limited field tests by Edgewood Arsenal, and submitted at the end of January 1977.

2. EXPERIMENTAL RESULTS

When the parts for one electrochemical cell were available, they were assembled and initial performance testing was begun. The sample gas heater and controlling thermistor were not included in the cell assembly, necessitating the closure of these lead-in ports. Initially the holes were closed with plastic tape placed on the outer surface of the cell. This proved insufficient to prevent significant leakage, so the holes were plugged with silicone rubber sealant. Leakage was no longer evident.

The cell, pumps, flowmeters, electronics, etc. were assembled as shown schematically in figure 1. Room air was passed through the cell at 2.0 l/min. and 0.1M tris buffer with 0.125 mN butyrlthiocholine iodide was pumped into the reagent inlet port. The anode was operated at a controlled potential 0.5 volts positive of the silver wire (Ag/AgI) reference electrode. The output signal indicating the current flow at the anode was displayed on a strip chart recorder.

A plain polyurethane foam pad was inserted in the cell and the system activated. Initially an extremely high and erratic current was observed. It was observed that the current fluctuations corresponded to large pulsations in the inlet gas flow rate. Every 4-7 seconds the gas flow would decrease markedly, momentarily stopping altogether. Upon inspection of the cell flow passages, it was discovered that silicone rubber sealant had intruded into the inlet port when assembled. Figure 2a shows the initial design and assembly method of this portion of the cell, wherein silicone rubber sealant was coated on the interfacing surfaces of the cell parts before assembly, the parts then being mated, held with screws and the rubber cured. Significant sealant entered the port, restricting fluid flow such that slugs of reagent solution would momentarily block gas passage during operation.

A collar was added to the cell design as shown in figure 2b, effectively preventing intrusion of the sealant during assembly and curing. Neither obstruction of the passages nor flow irregularities have been observed with the modified cell design.

The modified cell, when initially operated with a blank foam pad, exhibited a current of 100-150 μamps, unexpectedly high compared to the 1.0-1.5 μamps observed with earlier prototypes operated in the same manner. Scanning the anode vs. silver wire reference potential while monitoring

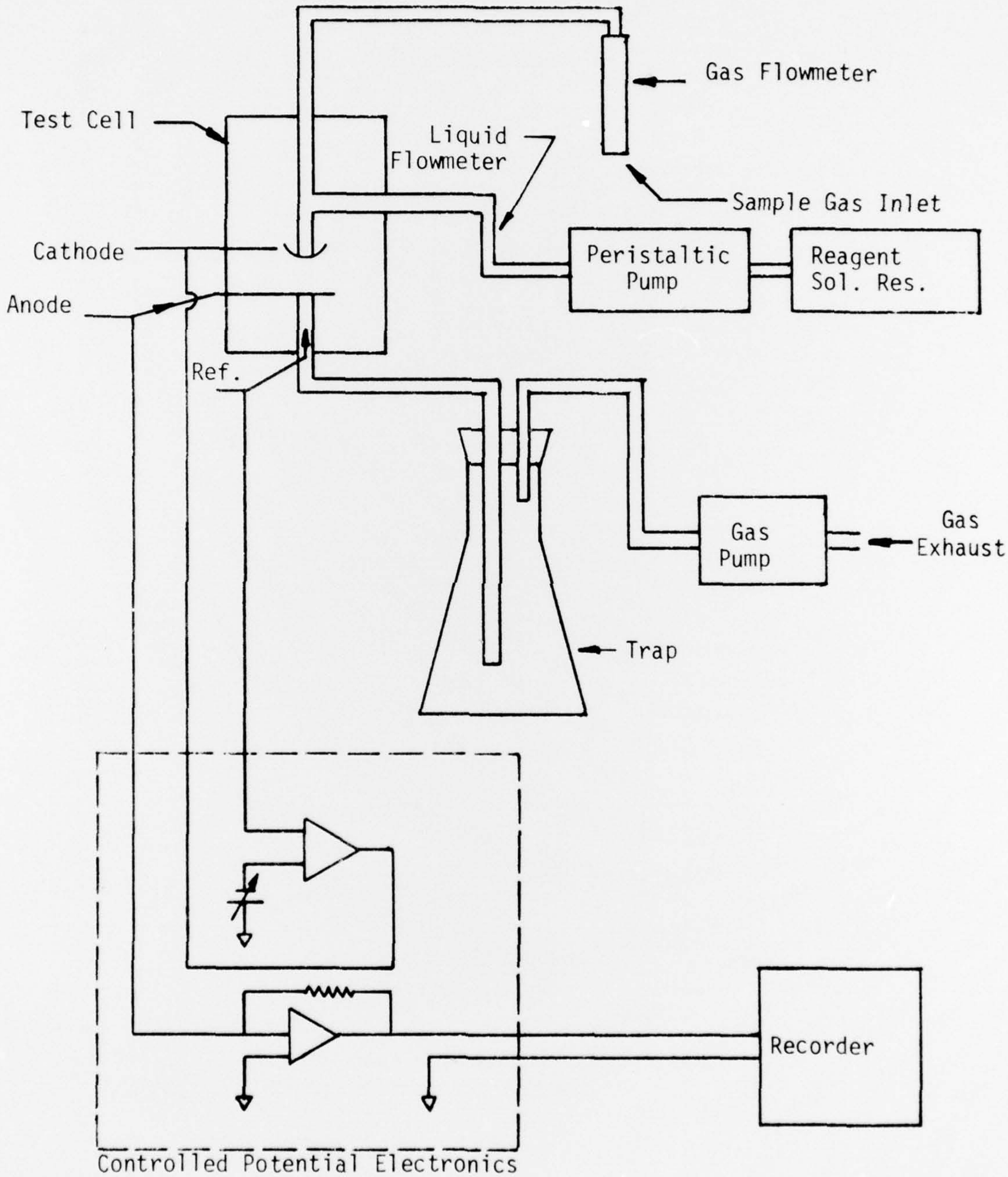

Figure 1. Schematic of Test System

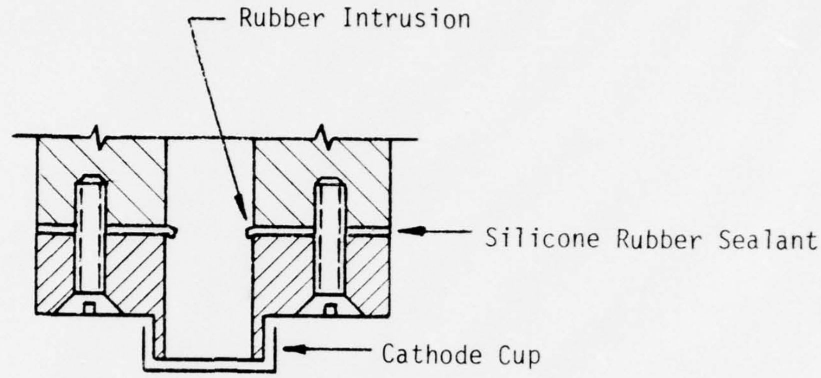

Figure 2a. Original Design Showing Area of Sealant Rubber Intrusion

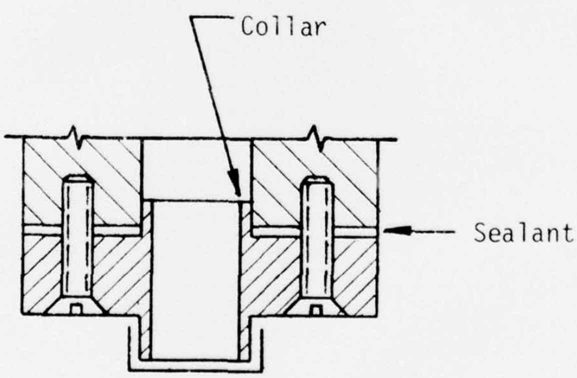

Figure 2b. Collar Added to Design to Prevent Sealant Intrusion

the current showed a typical current-voltage curve. However, there was an apparent displacement of the curve by +0.2 volts, indicating that the silver wire reference was not at the proper potential. This was confirmed by measuring the silver wire potential versus a standard calomel electrode with a pH meter. Whereas the silver wire in this system should be 0.15 volt negative of the calomel reference electrode, it was in fact 0.05 volts more positive.

A new supply of silver wire (99.9%) was obtained, the wire abraided as previously, washed, and assembled into a cell. Unlike the earlier case, the silver was then anodized briefly in situ using the internal cathode of the cell as the counter electrode. The potential of the new silver reference measured -0.15 volts versus calomel and the shift observed in the scan was, of course, corrected. The high current observed previously with a blank pad was attributable to iodide oxidation caused by the malfunctioning reference electrode.

When activated, the cell with its new reference wire behaved as expected. The current with a blank pad was initially 1.5 μamperes, decaying to 0.9 μamperes in the first 25 minutes of operation, and to 0.6 μamperes after 16 hours. With a starch entrapped enzyme pad, the current was initially 20 μamperes after 8 hours. This performance is typical of that previously observed with earlier prototype cells and starch-entrapped enzyme pads.

Having verified satisfactory performance of the cell with both active enzyme and blank pads, the latter simulating the limiting case of deactivation with anticholinesterase agent, further manufacture of additional cells and alarm components was continued according to schedule. No experimental evidence indicates problems which would jeopardize the achievement of the contract objective on time.

3. FURTHER WORK

The additional work to be completed under this contract includes the following:

(a) Final assembly of three enzyme alarm instruments.

(b) Laboratory testing of the assembled alarms.

(c) Such modifications as may be indicated necessary by the testing.

(d) Shipment of three enzyme alarms to Edgewood Arsenal for limited field testing.

(e) Any modifications agreed upon as a result of the limited field tests.

(f) Preparation, submission for approval, and publication of the Project Final Report.

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